

ABSTRACTS

Mechanical Properties of Films, Coatings and Interfacial Materials

June 27- July 2, 1999

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14. ABSTRACT The intended scope of the conference is to discuss research concerning the mechanical properties of thin films, coatings and interfacial materials. The program consisted of the following sessions: thin film delamination and deformation, stress and microstructural evolution, fracture and deformation of thin films and multilayers and properties and processing of multilayers and composites.					
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ORAL PRESENTATIONS

Mechanical Properties of Films, Coatings and Interfacial Materials
Oral Presentation

STRENGTHENING MECHANISMS IN THIN METAL FILMS ON SUBSTRATES

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Mechanisms responsible for the high strengths of thin films compared to their bulk counterparts are described. Yielding and strain hardening of thin metal films on substrates is studied by considering the motion of dislocations in thin crystalline layers. A simple model in which a pure edge dislocation is imagined to propagate in a film leaving a pure screw dislocation in its wake, at the film/substrate interface, is used to describe thin film plasticity. The method of images is used to determine the energy of the

THE INFLUENCE OF IMPERFECTIONS ON THE BUCKLING DRIVEN DELAMINATION OF THIN COMPRESSED FILMS

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ABSTRACT

The influence of prototypical imperfections on the nucleation and propagation stages of delamination of compressed thin films has been analyzed. Energy release rates for separations that develop from imperfections have been calculated. These demonstrate two characteristic quantities: a peak that governs nucleation and a minimum that controls propagation and failure. These quantities lead to two separate criteria that both need to be satisfied to cause failure. They involve a critical film thickness for nucleation and a critical imperfection wavelength for buckling. Implications for the avoidance of failure are discussed.

Mechanical Properties of Films, Coatings and Interfacial Materials
Oral Presentation

FAILURE MECHANISMS IN RESIDUALLY COMPRESSED FILMS

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A sequence of phenomena involved in the decohesion of residually compressed thin films and coatings is described. The process commences with the nucleation of an interface separation at an imperfection. Some typical imperfections are described and analysed. The separation extends and then buckles subject to a small scale buckling criterion, expressed through a buckling map. Thereafter, the buckle propagates stably until a kinking criterion is satisfied, whereupon a region of the film spalls away from the substrate. Each of these events is illustrated with examples based on thermally grown oxide films, DLC films or thermal barrier coatings.

**CRACKING AND DELAMINATION OF BRITTLE THIN FILMS WITH SMALL
SPHERICAL TIPPED INDENTERS.**

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The aim of this presentation is to critically appraise some of our recent work on the use of small, less than 10 micron radius, spherical tipped indenters with nanoindenters to initiate elastic/plastic/brittle response of thin films on ductile substrates. The advantage of spherical tipped indenters is that by appropriate choice of the ratio of indenter radius to film thickness one is able to change the dominant influence from that of the film to the substrate. Some examples to be considered include: TiN films on silicon, sapphire and stainless steel, DLC films on stainless steel, silica films on a polymeric substrate and ion beam modified surface layers of polymers. Cross-section observations of some of these systems along with complimentary acoustic emission measurements provide in some instances a clear indication whether cracking occurs upon loading or unloading. Numerical modelling of the stress fields indicates the extent of subsurface plasticity and likely location of film fracture.

References: Weppelmann & Swain, Thin Solid Films, 286 (1996) 111, Swain, Perry, Treglio, Elkind & Demaree, J.Mater.Res 12 (1997), Shiwa, Weppelmann, Munz, Swain & Kishi, J.Mater.Sci. 31 (1996) 5985, Thomsen, Fischer-Cripps & Swain, Thin Solid Films, (1998), Andersson, Toth, Gan & Swain, Eng. Frac. Mech. (1998).

MODELING OF HERTZIAN INDENTATION INTO LAYERED MATERIALS

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Starting with the approach of Hertz the problem of two spherical bodies in contact will be considered in this paper. The authors discuss the fundamental problems occurring as a result of the approximations being contained in the Hertzian theory with respect to its application to nanoindentation techniques.

After that a short introduction will be given to a mathematical method which allows one to find the complete analytical solution of any arbitrary contact problem for half-spaces with a layered structure. Hereby the model of a complete elastic homogeneous half-space covered with infinitely extended elastic plates ideally bonded to each other and to the half space as well will be the basis of our considerations. This model will be called

Mechanical Properties of Films, Coatings and Interfacial Materials
Oral Presentation

**STRUCTURE, COMPOSITION AND STABILITY OF INTERFACES BETWEEN
METALLIC THIN FILMS AND CERAMIC SUBSTRATES**

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Thin metallic films on ceramic substrates are important components for many technological important areas. Many properties of metal/ceramic systems are predominantly dictated by the properties of the interface between the two components. An understanding of the adhesion of the thin film requires knowledge of the structure, composition and bonding between metals and ceramics. Those parameters can be resolved for specific interfaces by high-resolution transmission electron microscopy (HRTEM), analytical electron microscopy (AEM) and detailed investigations of the energy-loss near-edge structure (ELNES) of the interfacial area.

Metal/ceramic components are often fabricated under elevated temperatures and may also be exposed to high temperatures under service. Under these conditions the stability of the interface plays a crucial role for the lifetime of the system. The morphological instability as well as chemical instabilities have to be considered.

In this paper detailed studies on the Cu/ α -Al₂O₃ interface and the Pd/SrTiO₂ interface will be described and discussed.

Mechanical Properties of Films, Coatings and Interfacial Materials
Oral Presentation

**A MECHANISTIC STUDY OF THE EFFECT OF INTERFACE MORPHOLOGY ON
THE OXIDATION- INDUCED STRESSES IN A THERMAL BARRIER COATING
SYSTEM**

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The oxidation-induced stresses which are known to control the interface degradation and eventual failure of ceramic-metal interfaces at high temperatures are calculated using a constitutive framework coupled with the diffusion of oxidant species through a multi-phase solid. The proposed internal variable constitutive model accounts for the phase transformation associated with the oxidising phase, and incorporates the effect that the local volumetric expansion of the newly formed oxide has in the generation of inelastic volumetric strains and stresses. The formulation is implemented into the finite element method and used to investigate the effect of the ceramic-metal interface morphology on the evolution of the interfacial stresses induced by the continuous oxidation of a typical plasma-sprayed thermal barrier coating system.

**CONDITIONS TO AVOID CRACKING AND DEBONDING IN INTEGRATED
CIRCUIT STRUCTURES**

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When the fracture mechanics is applied to a bulk structure, an essential input is the information of a pre-existing crack, i.e., its location, size, and orientation. This information is impossible to obtain in practice for an integrated circuit. We will adopt a different strategy based on two facts about an integrated circuit. First, high tensile stress is generated by internal misfit, and is therefore confined in small regions of size comparable to the feature size. Second, the fabrication process is controlled down to individual features, so that the pre-existing cracks are expected to be smaller than the feature sizes. Instead of considering one pre-existing crack, we consider all possible pre-existing cracks. We then identify the worst case, namely, the crack with the largest energy release rate and the smallest fracture energy. If this crack cannot grow, nor can any other cracks. Such a no-cracking condition needs no information on pre-existing cracks; rather, it depends on parameters that defines a circuit structure, such as the feature size and the aspect ratios of the geometry. Examples will be given to illustrate the approach, including cracking induced by thermal expansion misfit and electromigration.

Mechanical Properties of Films, Coatings and Interfacial Materials
Oral Presentation
Poster Presentation

ADHESION OF GOLD-CHROME MULTILAYER FILMS IN HYBRID MICROCIRCUITS

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Interface adhesion is an important factor controlling the performance and reliability of thin films. It is particularly important in hybrid microcircuits where adhesion along interfaces between gold conducting and chrome adhesion layers can change markedly over time through diffusion, segregation, and phase transformations. Nevertheless, the effect of these changes on mechanical reliability is known only by relative comparisons due to a lack of quantitative test techniques. We have therefore begun a systematic study to determine the relationship between structure, properties and adhesion of gold, gold-on-chrome, and chrome films on sapphire substrates used in hybrid microcircuits. These films were sputter deposited following production procedures. Following deposition, elastic and plastic properties of the film systems were determined using nanoindentation tests coupled with direct measurement of contact areas. A tantalum nitride overlayer was then sputter deposited onto all film systems to uniformly stress the films and help drive fracture. These stresses triggered interfacial delamination and blister formation in the gold and gold-on-chrome films while additional stresses from nanoindentation were required to induce delamination and blister formation in the chrome films. Using the mechanics based models of Hutchinson and Suo for blister formation modified for multilayer effects, elastic properties from nanoindentation, and measured blister heights and widths, residual stresses and interfacial fracture energies were determined for all three film systems. The results showed that the gold films formed the weakest bonds with values near those for van der Waals forces while chrome films formed the strongest bonds with values near those for metallic bonding. Gold-on-chrome bond strength fell somewhere between. In this presentation, the results will be presented and used to show how interface composition and structure affects film adhesion and the concern that gold-chrome interface failure poses for long term mechanical reliability of these film systems. This work supported by U.S. DOE Contract DE-AC04-94AL85000.

EFFECT OF PLASTICITY ON DELAMINATION

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A number of techniques have been developed to measure the adhesion between a substrate and a thin coating or laminate. Among these is the peel test. If the laminate deforms in a purely elastic fashion, the peel force is a direct measure of the interfacial toughness. However, if the laminate deforms in a plastic fashion, additional energy is absorbed which dominates the peel force. An embedded-process zone (EPZ) model, in which the interfacial parameters consist of a peak cohesive stress and an intrinsic interfacial toughness has been used to explore the mechanics of the peel test. The predictions are compared with experiments performed on model systems consisting of sheet metal bonded with an adhesive.

Simple beam theory can be used to calculate the energy absorbed by plastic bending during a peel test. However, these energy-balance approaches ignore the effect of the cohesive stress which plays a crucial role when large-scale plastic deformation occurs. This can be illustrated by using an EPZ model to predict the peel force. Such an analysis demonstrates that the peel force increases with the thickness of the laminate. When the thickness is relatively large, beam-bending analyses result in a lower-bound to the peel force. The EPZ model shows that the crack-tip stresses associated with the cohesive zone introduce a hydrostatic state of stress which restricts plastic deformation, and invalidates the use of simple-beam theory.

A model system consisting of aluminum bonded by a commercial adhesive has been used to verify the EPZ model. The interfacial parameters for this model system were determined in previous experiments and incorporated into an analysis of the peel test without further modification. A comparison between experimental measurements of the peel force and the numerical predictions of the EPZ model showed excellent agreement, both in the initial stages of peeling and for the steady-state values of the peel force.

FINITE ELEMENT MODELLING OF CONTACT STRESSES IN COATED SURFACES

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This paper describes the development of a finite element model of the contact between a spherical indenter and a plane, coated surface. The model is unique in allowing the specification of realistic mechanical properties for the indenter, coating, and substrate, whereas previous models have tended to use either a rigid indenter or an imposed pressure distribution.

It is shown that earlier models have substantially underestimated some of the contact stresses that could cause coating failure.

It is also shown that, by using the model and the results of indentation experiments, it is possible to determine values for the coating hardness and modulus that are not influenced by substrate properties.

STRESS EVOLUTION AND MICROSTRUCTURE IN MULTILAYER FILMS

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The growth processes during the initial stages of growth often result in stresses in the growing film. These processes, including island growth and coalescence, epitaxy, defect production and annihilation, interface formation and compositional intermixing determine the properties and microstructure of the film. The resulting stress can be substantial and important for thin film and multilayer applications. Moreover, in-situ measurement of stresses can be used to monitor growth processes and their dependence on processing parameters.

This talk will present the results of in-situ measurements of stress in several thin film and multilayer systems. The growth of Pt on glass occurs by an island growth process and the stress behavior reflects the island growth with a compressive behavior and island coalescence with a tensile excursion. In the Mo/Si multilayer system, the initial stages of growth of Mo on Si and Si on Mo are characterized by large stress transients. These transients (compressive in Si and tensile in Mo) are related to the diffusional asymmetry in the interface intermixing during growth. For fcc metal superlattice structures the initial layer stress is strongly dependent on the size and surface energy mismatch with the layer upon which it is growing. For example in the Pd./Pt system, Pt grown on thick Pd layers shows a coherency stress which is absent in Pt grown on thin coherently strained Pd. In the Ag/Pd system, Ag shows a sharp compressive drop for the first monolayer consistent with coherency and surface stress differences. Subsequent Ag growth is completely relaxed. This behavior is consistent with Stranski-Krastanov growth mode.

Effects of Viscoplasticity on Perimeter Stress Distributions and Reliability in Multilayered, Ductile/Brittle Electronic Packages

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Material systems comprised of ductile/brittle multilayers are ubiquitous within the world of electronics, starting with the multiple layers on devices, continuing through the interconnections, to the final assembly onto substrates. The transfer of stress across these interfaces represents a crucial design issue over several length scales. This is a result of the dominant role of the magnitude and time-dependence of mechanical stress and strain on the *reliability*. However, exact knowledge of the in-situ thermo-mechanical residual stress that is generated by various interfacial materials in multilayered electronic systems, the effects on reliability and the underlying physical basis has been elusive.

Here, the results from a recent series of detailed experimental and theoretical analyses will be reviewed that have revealed, with unprecedented resolution, the in-situ spatial and temporal distribution of thermo-mechanical residual stress develops within an elastic layer joined to an elastic substrate with an elastic or viscoplastic layer. Specifically, experiments have been conducted with silicon bonded to a variety of substrates utilizing four solder/braze materials, and the distributions of stress within the silicon have been measured utilizing piezospectroscopy with 1 micron and 20 MPa resolution. Special emphasis is devoted to examining the stress fields in the vicinity of the free surfaces and edges. These results are analyzed within the context of simple, micromechanics models and found to yield excellent agreement with the theoretical predictions. Furthermore, the effects of creep relaxation on the redistribution of stress will be demonstrated through direct experimental observations, and the effects of the layered morphology on the creep rate within the adhesive will be described. Finally, the effects of thermal cycling on the distribution of stress, fatigue crack initiation and growth will be presented and analyzed. In conclusion, the implications of these results on the design and reliability of multilayered systems will be summarized.

INTERFACE EVOLUTION AND THE DELAMINATION AND SPALLING OF OXIDE SCALES AND THERMAL BARRIER COATINGS

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Thermal Barrier Coatings (TBC's) and oxide thin films designed to protect metallic components in high-temperature, oxidizing atmospheres are susceptible to failure by delamination, buckling and spalling. Thermally grown oxides (TGO's) and TBC's fabricated by electron beam physical vapor deposition (EBPVD) generally fail by delamination at the metal/oxide interface with subsequent kinking of the interfacial crack through the oxide layer(s). Coating failure is motivated by residual stresses that develop due to oxide growth and thermal expansion mismatch with the underlying metal. The thermo-mechanical behavior of the material system is governed by the fracture characteristics of the metal/oxide interface and the oxide layer(s). Fracture-mechanics-based test methodologies have been developed for quantitative studies of the fracture characteristics of the oxide layer(s) and the metal/oxide interface. The techniques have been implemented to study mechanistic aspects of interfacial failure and the evolution of TBC systems during high-temperature, oxidizing atmosphere exposure. Particular attention is focussed on morphological development of the metal/oxide interface during oxidation, as morphology plays a key role in the mechanics of interfacial cracking and damage coalescence. Studies on commercial TBC systems are combined with analytical treatments, numerical simulations and experiments on materials with artificially induced morphology to elucidate the effects of morphological features on the nucleation of interfacial separations and the propagation of damage. Implications of the interface morphology, bond coat chemistry and structure, and oxide growth mechanisms for the spalling failure and general thermo-mechanical performance of TBC's will be discussed.

THE ADHESION OF ALUMINA FILMS TO METALLIC ALLOYS AND COATINGS

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The adherence of protective oxide scales to alloy substrates is governed by the stored elastic energy in the scale which drives delamination and the fracture resistance of the alloy oxide interface. Clearly, any modifications to the alloy or the exposure environment which decreases the former or increases the latter will improve the durability of a given system.

The stored elastic energy is determined by the stress level in the scale and the scale thickness. The stress state in the scale is determined by stresses which arise during the oxide formation (Growth Stresses), stresses produced during temperature changes as the result of thermal expansion mismatch between the oxide and the alloy (Thermal Stresses), and any stress relaxation which occurs as the result of creep of the scale or alloy.

The fracture energy of the interface is a function of the composition at the interface, the microstructure in the interfacial region, and the composition of the exposure environment.

This presentation will focus on the results of studies of a variety of alloys and coatings, all of which form continuous alumina scales, in which it has been attempted to evaluate the effects of various alloy and exposure parameters on the stress state in the scale, the microstructure of the alloy/oxide interface, and the fracture resistance of the interface. The alloy parameters include alloy type, sulfur content, reactive element content, and presence of a zirconia overlayer (thermal barrier coating). The exposure parameters include oxidation temperature, temperature profile during cooling, and water vapor content of the atmosphere.

MECHANICAL BEHAVIOR OF THERMAL BARRIER COATINGS UNDER THERMOMECHANICAL LOADING

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The mechanical behavior of multilayer thermal protective coatings on Ni-based superalloy substrates under combined thermal and mechanical loads is studied using a finite element analysis approach. The thermal barrier coating system under investigation consists of a bond coating, a thermally grown oxide (TGO), and a ceramic plasma sprayed top coating. All materials are treated as isotropic linearly elastic - perfectly plastic, except the ceramic top coating that is considered as perfectly elastic, while special care has been taken in order to incorporate accurate values for the material properties required. The influence of interfacial topology is also examined by comparing the stress states at the vicinity of ideally rough bond coating / TGO and TGO / ceramic top coating interfaces, with the stresses generated along planar interfaces. The analysis demonstrates that, under the examined thermomechanical loading conditions, the ceramic top coating is mainly under compression, while significant tensile stresses develop during the heating/mechanical loading and cooling/mechanical unloading segments of the loading profile. The very low yield strength of the bond coating at elevated temperatures is responsible for the plastic deformation of this layer that ultimately leads to the development of extensive residual tensile stresses within the bond coating. Finally, the TGO experiences significant tensile stresses throughout loading, comparable to values measured experimentally. The presence of a rough interface generates significant shear and radial tensile stresses at the bond coating / TGO, and TGO / ceramic top coating interfaces that may be responsible for interface decohesion. The results of the analysis are discussed in terms of the spallation and failure of thermal barrier coatings: the thermal and loading conditions that promote spallation are identified and failure mechanisms are described.

DECIPHERING INDENTATION LOAD-DISPLACEMENT CURVES

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Measurement of thin film mechanical properties by load and depth sensing indentation methods, often referred to as nanoindentation, is based on load-displacement data obtained as an indenter is driven into and withdrawn from a material. For sharp indenters like the Berkovich three-sided pyramid used frequently in nanoindentation studies, indentation loading curves are usually well-described by the simple power law relation $P = Bh^2$, where P is the load, h is the penetration depth, and B is a constant related to the geometry of the indenter and the elastic and plastic properties of the material. The dependence of the load on the square of the penetration depth is a natural consequence of the self-similarity of the pyramidal indenter, as it would also be for indentation by a rigid cone. During unloading, the load-displacement behavior is considerably more complicated due to the complex elastic and plastic processes which determine the shape of the permanently deformed surface after the indenter is withdrawn. Despite this, experiments have shown that the unloading curve can also be described by a power law function of slightly different form. The relation $P = A(h-h_f)^n$ is often used, where h_f is the final depth after complete unloading, and A and n are material constants. However, unlike the loading curve, the power law exponent for the unloading curve is not fixed at an integral value, but varies from material to material in the range $n=1.2-1.6$. Simple models based on observations from finite element simulations of indentation by a rigid cone are presented which provide a physical explanation for these behaviors. The models also provide a means by which the material constants appearing in the power law relations can be related to more fundamental material properties such as the elastic modulus and yield strength.

**PLASTIC DEFORMATION IN DIFFERENT TEXTURE COMPONENTS
IN COPPER THIN FILMS**

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Thin films on substrates generally support very high stresses due to differential thermal expansion. These stresses may be much higher than those that would be predicted on the basis of the behavior of bulk metals due to dimensional and microstructural constraints on dislocation motion in these materials. These stresses are often investigated by measuring the changes in curvature that are induced in a film/substrate package by changes in temperature. The stresses that are measured in such an experiment represent average values. Although these values are closely representative of the actual stresses in elastically isotropic films, the local stresses can vary dramatically from these average values for particular texture components in elastically anisotropic films. Cu films in particular, often have grains with 111 and 100 planes parallel to the film plane as the primary texture components. Precisely these components have the highest (261 GPa) and lowest (115 GPa) biaxial elastic moduli, respectively, of the crystal orientations in Cu. Thus, when a nominally uniform strain is applied by differential thermal expansion, the stress states will be markedly different in these different texture components. The stress states in various texture components of Cu films on Si substrates during thermal cycling have been examined using x-ray strain measurements. The results show that different texture components indeed react differently, but in surprising ways. For example, the behaviors in compression upon heating and in tension upon cooling are quite different from one another. These results suggest that the plastic deformation of a copper film depends in a complex way on the deformation of the individual texture components. Knowledge of the distribution of stresses is required in order to understand stress-driven processes like electromigration, void formation and decohesion.

MEASUREMENT OF STRESS IN FILMS USING OPTICAL SPECTROSCOPIES

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Optical spectroscopic techniques, such as Raman or luminescence, in which characteristic frequencies depend on lattice strain, are useful methods for studying stress distributions in films and coatings. They are non-destructive, quick and potentially have high spatial resolution when used in conjunction with an optical microprobe. When the stress state in the film is known and the stresses are uniform within the volume of material analysed by the microprobe, the measurement of stress is straightforward. However, if either of these conditions is not met then the optical response is difficult to interpret in terms of the stress distribution.

One method of approaching this problem is convenient if the stress distribution can first be estimated by some other means, such as finite element modelling (FEM). Here we show how the stress distribution can be convoluted with the optical characteristics (focusing parameters) of the incident beam and the solid medium (refractive index and absorption coefficient). The focusing parameters can be determined by suitable measurements on bulk specimens. It is also shown that the widely used method of averaging the line shift over the analysed volume is not correct and can lead to large errors. The method is illustrated by application to Raman measurements of quantum wires and LOCOS structures on silicon substrates.

A second method is also described that can be used when the stress distribution is unknown. This involves measuring a series of spectra using light with different optical absorption coefficients. These can then be mathematically transformed, using a Laplace transformation, to give the stress distribution. Under appropriate conditions, focusing the incident beam at different positions below the specimen surface produces changes in the distribution of light intensity that simulate the effects of changing the optical absorption coefficient. This is proposed as a convenient alternative to using light of different frequencies.

**CRACK NUCLEATION AT BIMATERIAL CORNERS: A FRACTURE PROCESS
ZONE APPROACH**

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The purpose of this work was to study the severity of bimaterial corners with respect to the corner angle and formulate a crack nucleation criterion that predicts the failure loads for different corner angles. Analytical solutions were obtained for a general bimaterial corner with varying material properties and corner angles. The stress singularities and the stress intensity factors were obtained. It was seen that the stress intensity factors varied with the corner angles and were not in one-to-one correspondence with the stresses and hence were not adequate to quantify the severity of the corner by themselves. A series of experiments were conducted where an aluminum-epoxy bimaterial specimen was loaded under 4-point bending and the displacements were measured using moiré interferometry. Numerical analysis was conducted with a rigid interface and the displacements compared with the measured ones. The displacements differed with each other, especially near the corner and along the interface. A Cohesive Zone Model (CZM) was incorporated in the numerical analysis and, after calibration with an interface crack, was found to yield displacements for other corners that were in good agreement with the measured values. The predicted failure loads for all corners were also in good agreement with the experimental results. Finally, for design purposes, a simple parameter was proposed to predict the failure load of a bimaterial specimen with an arbitrary corner angle based on the failure load of a bimaterial specimen with an interface crack.

ANALYSIS OF THE MECHANICAL STABILITY OF FILM ON SUBSTRATE SYSTEMS, FROM MICROMECHANICAL EXPERIMENTS

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Nowadays complex film on substrate systems are used in different technological domains. For example: thin layers of materials offering a high reflectance are assembled for mirrors intended for laser guidance; also thin layers of isolating and conducting materials are superimposed in microelectronic devices. In both cases, even if optical or electrical considerations remain predominant in the designing of the above mentioned devices, their reliability and performances can be limited because mechanical problems.

For a better determination and understanding of the properties of the thin films consisting a system, several micro mechanical tests have been developed in the last years. The most relevant of them are: the depth sensing indentation and the bulge test, which allow an accurate determination of the elastic properties of a film. However, in order to understand how a film on substrate system will degrade, then mechanically fail, it is essential to investigate it's mechanical response when submitted to external stresses. To perform this sort of investigation, we developed micromechanical devices, which were adapted in a SEM.

Then, a contineous observation of the evolution of damage with the imposed strain (for example from the cracking of a film, to it's debonding), will allow a precise analysis of the mechanical stability of the investigated system. Besides, the experimental re sults can be confronted to different theoretical models, from which theoretical stress redistributions during cracking can be established, and intrinsic film parameters determined.

**INFLUENCE OF INTERFACE STRUCTURE AND DEFECTS ON FRACTURE
PROPERTIES OF METAL TOUGHENED CERAMICS**

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The fracture properties of ceramics toughened by the addition of a ductile metal are strongly influenced by interfacial debonding that can occur between the metal ligaments and the ceramic matrix. Debonding controls the volume of deforming metal and the degree of elastic constraint exerted on the metal by the ceramic matrix. The volume of metal undergoing plastic deformation and the metal flow characteristics due to constraint during fracture effect the fracture resistance (R-curve) behavior of the composite. Control of the interface structure and defects is necessary to control debonding and hence tailor the fracture behavior of ceramic metal composites. The structure of the metal-ceramic interface is effected by the processing technique chosen to produce the composite. This paper relates the metal-ceramic interface structure to processing technique and the resulting fracture properties. Interfaces and defect structures from Al/Al₂O₃ composites produced by directed metal oxidation, reactive metal penetration and pressure assisted infiltration are correlated with fracture properties and fractography. Defect spacing at the metal ceramic interface is related to the degree of debonding that occurs during fracture. Closely spaced defects can act as void nucleation sites for a ductile debonding mechanism based on microvoid coalescence with the metal. The defect spacing to induce ductile debonding appears to be related to the size of the metal phase. Model experiments on Al₂O₃/Al/Al₂O₃ sandwich specimens with a range of defects confirms the influence of defect spacing on void nucleation in the metal. This work shows that although very strong bonds can occur between Al and Al₂O₃, the amount of debonding can be controlled by an array of defects at the interface.

ESTIMATION OF FRACTURE TOUGHNESS OF THIN FILMS BY VICKERS INDENTATION CRACKING

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A key parameter, which is responsible for the performance of a film-coated-substrate system in any mechanical application, is the fracture toughness of the film material. In addition, residual stresses are generally produced during the synthesis process in both of the film and substrate and it is important to elucidate the influence of the residual stresses on the fracture behavior of the film/substrate systems. It is well known that radial cracks are formed by impressing a Vickers indenter on the surface of a brittle material, and that the fracture toughness, K_{IC} , is given by $K_{IC} = xP/C^{3/2}$, where x is the material-dependent factor, P is the indentation load, and C is half the crack length. The crack size also changes depending on the residual stress. Therefore the indentation cracking is expected to give information about both residual stress and fracture resistance of thin films coated on brittle materials.

In the present study, we will propose a fracture mechanics model of Vickers-indentation cracks formed in thin film/brittle substrate systems. In order to analyze the influence of residual stress and fracture resistance, indentation radial cracks were made in thin-film-coated Si single crystals and the change in crack-size and crack-geometry was monitored as a function of indentation load. The crack-size of the film-coated Si crystals did not obey the $P-C^{3/2}$ relation. Further the crack-geometry was neither semi-circular nor could the geometry be simply anticipated from the residual stress distribution before indentation cracking. According to these results, we postulate (1) that net stresses acting on the crack fronts in the film and substrate are composed of the residual stresses before indentation cracking and the traction stresses on the crack fronts, (2) that the crack geometry is not semi-circular, and (3) that the minimum energy configurations of the crack fronts are given by an equilibrium condition of crack driving force between the film and substrate. The model established upon these assumptions enables the evaluation of the fracture toughness and residual stress of thin films. Recent results will be shown and discussed.

**KINETIC-BASED INVESTIGATIONS OF STRENGTHENING MECHANISMS IN
NANOLAYERED COMPOSITE THIN FILMS**

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Strengthening in nanolayered composite thin films has been attributed to a number of factors including the layer interface Hall-Petch, modulus mismatch, coherency strains, and layer grain size. In general in order to identify and distinguish between these mechanisms, the effects of layer thickness on hardness are examined. Models predict that the hardness will be the sum of two independent components, one depending on layer thickness. Here, we devise an activation analysis of nanoindentation experiments to investigate hardening mechanisms. The analysis consists in examining the effects of layer thickness on hardness H and rate sensitivity of the hardness. These studies are carried out in conjunction with microstructural investigations. Nanoindentation experiments have been performed on Cu/Nb, Cu/Ni, and SiGeC/Si multilayers, in which Hall-Petch, modulus mismatch, and coherency strain effects, respectively, are important.

DEFORMATION AND FAILURE OF METALLIC THIN FILMS CAUSED BY THERMAL STRESS

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Prevention of stress-driven failure is an important technological issue in metallic interconnect lines in silicon device. Failure occurs during processing at high temperatures and during device operation. The former accompanies stress migration failure leading to hillock formation. The latter accompanies electromigration failure leading to hillock and void formation. Furthermore, new device designs require a smaller size and a larger current density. This may induce noticeable temperature rise that accompanies thermal stress cycling and cyclic fatigue failure during operation. There have been intensive research efforts to understand the mechanism of these failure modes. On the other hand, interaction of various factors causing the change in thermal stress may hamper a clear understanding of failure mechanisms and underlying deformation mechanisms. In this paper, examples of stress migration failure and cyclic fatigue failure are presented in Al/Si. Recent results in Cu/Si are also presented and compared with Al/Si.

In the Al/Si system, stress migration was investigated by annealing as-deposited films. Quantitative agreement in the magnitude of stress relaxation was obtained in stress relaxation between experimental and theoretical based on the volume change caused by pushing out of hillock to the surface. Cyclic fatigue failure was investigated in annealed films of Al. In the annealed films, stress relaxation by hillock formation and by grain growth could be eliminated from consideration, thus, revealing the effects of thermal cycling. During thermal cycling between RT and 450 °C, residual stress increased up to the 4th cycle, followed by a rapid decrease. The increase was caused by accumulation of dislocations. The decrease was caused by the formation of grain-boundary cracks. Based on the analysis of stress relaxation data, dislocation glide was found to be a major deformation mechanism. The analysis was in general agreement with a calculated deformation mechanism map.

Mechanical Properties of Films, Coatings and Interfacial Materials
Oral Presentation

ASPECTS OF PLASTICITY IN METALLIC MULTILAYERS

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The properties of metallic multilayers is influenced by the scale, the epitaxy, the residual stresses and the interface mixing. In attempting to produce models of mechanical behaviour, one needs to consider factors such as difference in shear moduli, internal stress and detailed nature of interface. Thus a variety of systems can be used to control those factors such as Cu/Ni, Cu/Cr, Cu/Ag and Cu/Nb.

In order to deduce the mechanical properties, dedicated experiments were designed: injection of dislocations from a single crystal substrate into the multilayer, penetration by an indenter, tensile tests on free standing films, and measurements of the residual stresses.

The results on both mechanical tests and detailed characterization of microstructure will be integrated within models describing the plasticity of multilayers

Interface stress of nanocrystalline materials

Verbal

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Nanocrystalline (nc) materials are characterized by a high density of internal interfaces (grain- or phase boundaries). Therefore, the pressure that is exerted on the nanometer-sized crystallites through the presence of interfaces can be determined by means of careful x-ray residual stress analysis. With this result, we can calculate the mean interface stress when employing thermodynamic identities between pressure and elastic moduli as well as using the concept of effective elastic moduli. For fcc materials, we find the interface stress being positive and of the order of 1J/sqm, whereas, ceramic nanoscaled materials exhibit evidence for negative interface stress.

THE ROLE OF SURFACE FILMS IN THE TRIBOLOGICAL BEHAVIOUR OF MELT OXIDISED Al_2O_3 - SiC - Al COMPOSITES

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Alumina- aluminium matrix composites have been fabricated with a range of SiC particulate dispersions and tested for their wear resistance under adhesive and abrasive sliding conditions. It has been shown that the presence of the soft metallic phase brings about high wear resistance and low friction owing to the formation of a tribo-chemical surface film, particularly under high loads and speeds in unlubricated conditions. Against steel, at speeds of up to 10 m/s, the composite develops a surface layer that consists of Fe_3O_4 and Al owing to the high surface temperatures that permit softening or melting of the residual alloy in the composite. Low and cyclically varying wear rates and friction coefficients follow from the formation and removal of this tribo-film which leads to low contact stresses. Similarly, under abrasive conditions against glass-bonded SiC discs, a metal containing debris blunts the grinding grit and reduces the wear rate. Despite the lower hardnesses, by a factor of 2-4, compared to ceramics such as zirconia-toughened alumina and silicon carbide, the present composites offer a comparable or lower wear resistance. Simulated mechanical seal tests against graphite reveal that extremely low friction coefficients are attained, suggesting that the present composite may have applications in bearings.

**POSTER
PRESENTATIONS**

Thermally-Dependent Mechanical Properties of Arc-Evaporated Cr-N Coatings

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The temperature dependence of the mechanical properties of arc-evaporated Cr-N coatings has been investigated. These properties include the elastic, elastic-plastic and fracture responses, which have been measured on both as-deposited and tempered coatings using a combination of scratch, nanoindentation, abrasive wear and surface acoustic wave (SAW) experiments.

The coatings were deposited onto tool steel substrates at two bias voltages, 50V and 300V, and then subjected to moderate tempering in the range 280 - 650 C for 4.5 hours. The deposition conditions were chosen, based on results from previous studies, to provide coatings with similar thickness and levels of residual stress, but different degrees of defect stability with temperature.

The tempering was found to change the mechanical response of the coatings dramatically. The dispersion curves, as measured by SAW, increased with tempering temperature, which is interpreted as an increase in the ratio between the transverse elastic modulus and density of the coatings. The hardness of the coatings was found to change by as much as 30% during tempering. Furthermore, the hardness variations were bias dependent, as the 300V sample displayed a systematic decrease while the 50V sample displayed a local maximum with increasing temperature. Tempering also affected the scratch resistance of the coatings, as measured by the critical loads for coating flaking and substrate exposure. These results were further investigated by observations of the scratch tracks, and are discussed in terms of different fracture mechanisms occurring during the scratch tests for different tempering values. In addition, changes in the wear behavior with tempering, as determined by pin-on-disk testing, are presented.

In addition to evaluating the mechanical properties, the microstructure, residual stress, and texture of the coatings were characterized using X-ray diffraction and cross-sectional transmission electron microscopy. The tempering was found to systematically decrease the lattice defect density in the coatings. Furthermore, at higher temperatures the grain structure was observed to transfer from pronounced columnar to partly equiaxed, and a distinct band was formed at the coating/substrate interface, indicating diffusion processes in this region. The measured residual stress was compressive in all coatings, and decreased with tempering, from approximately 4 GPa in the as-deposited coatings down to approximately 1 GPa. The preferred orientation in the growth direction was [110] for both bias values, but more than twice as strong for the 300V sample, and this difference persisted with tempering.

The relationships between these mechanical and microstructural properties are discussed in terms of current models for elastic-plastic and fracture behavior in thin films. Emphasis is placed on the roles of residual stress, defect density and microstructure.

DAMAGE DUE TO LASER IRRADIATION OF THERMAL BARRIER COATINGS - FRACTURE MECHANICAL MODELLING

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Cyclic surface heating by a moving laser beam proved to be a suitable test for the performance of thermal barrier coatings (TBCs). ZrO₂-TBCs prepared by means of electron beam vapour deposition were subjected to such tests. The temperature field was controlled by means of a high speed pyrometer. The maximum temperature T_{max} and the number of cycles N served as the principal parameters of the tests.

Subsequently the damage was investigated by microscopy and by metallographic cuts. Damage was found to develop in several stages with increasing numbers of cycles: vertical cracking --> delamination cracks --> local blistering --> local spalling.

Unstable delamination revealed itself by a jump in the recorded temperature vs. time curve. The regions in the (T_{max} , N)-plane where the different stages of damage have been observed make up a damage map.

This damage scenario can be understood by means of creep and shrinkage due to sintering of the TBC. Progressive shrinkage during thermal cycling increases the energy release rate for vertical cracks which subsequently turn into delamination cracks. Unstable and stable propagation of delamination cracks is modelled by means of the finite element method (FEM) assuming increasing shrinkage in the TBC.

A decrease of interface fracture toughness due to aging has been measured by means of a 4-point bending test modified by a stiffening layer. Correlations with laser heating damage will be discussed.

Cyclic surface heating by laser irradiation enables us to evaluate the damage resistance of TBCs. Hence, such tests are a useful means for investigating the influence of aging on the performance of TBCs.

FATIGUE TESTING OF THERMAL BARRIER COATINGS UNDER REALISTIC TESTING CONDITIONS

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Thermal barrier coatings (TBC) on components with inner cooling have a high potential with respect to higher turbine gas temperatures and hence reduced fuel consumption of turbine engines. This potential can be exhausted only, if these coatings can reliably withstand their extreme thermal and mechanical fatigue loading. Failure of the TBC at increased gas temperatures would lead to failure of the whole component.

Demonstration of TBCs reliability requires realistic testing, to make sure that lifetime determining mechanisms under testing conditions are the same as in real engines. Therefore a thermal gradient mechanical fatigue (TGMF) testing equipment was developed, which allows simultaneous mechanical and thermal loading, including thermo-shock. For heating the radiation of four bulbs is focused on the specimen by elliptical mirrors. For rapid cooling to simulate thermo-shock sliders with integrated vents for cold air can be closed. Thermal gradient was imposed by inner air-cooling of hollow cylindrical specimens, allowing higher temperature in the ceramic topcoat than in the metallic substrate.

The loading test-cycle was selected on the basis of a normal flight of the engine of an civil airliner with start, cruise and landing. The thermo-mechanical load was adjusted under the assumption that the metal blade or specimen substrate respectively determines thermo-mechanical loading conditions imposed on the TBC. For the specimen substrate without TBC the thermo-mechanical load was chosen so that the life of the specimen without TBC is matched to that of the blade in a turbine. Therefore the lifetime limiting thermo-mechanical loading conditions should be equivalent. Inner cooling allows higher temperatures within the TBC than in the metallic substrate while the strain is imposed by the metallic substrate. Depending on adjustment of loading conditions for specimens with TBC the TBC-lifetime for different service conditions will be determined.

MICROSCRATCH TEST ADHERENCE STUDY OF AMORPHOUS SILICA AND SILICON FILMS DEPOSITED ON STAINLESS STEEL BY P.E.C.V.D.

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Abstract

Amorphous silica and hydrogenated silicon films 0.5 μm thick were deposited by P.E.C.V.D. Two different systems were used, a micro-wave plasma reactor (2.45 GHz) with I.D.E.C.R. configuration for silica and a R.F. reactor (13.56 MHz) for amorphous silicon. Pretreatment of stainless steel substrates (316L) with hydrogen or nitrogen plasmas were in situ achieved prior to the film growth.

Adherence was tested by micro-scratching using a MST-CSEM[®] system equipped with optical microscope, acoustic emission sensor and tangential force sensor. Two critical loads were valued from the optical observations according to the measurements of acoustic emission and tangential force. Determination of the critical loads and analysis of tangential force curves were reliable. This test has showed the function of the pretreatments considering the reactor configurations and allowed to compare their effects on the adherence of coatings.

MICROSCRATCH TEST STUDY OF TiN FILMS DEPOSITED ON SILICON BY R.T.L.P.C.V.D.

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Abstract

TiN films were deposited on silicon wafers at 800°C by R.T.L.P.C.V.D. with $\text{TiCl}_4 + \text{NH}_3 + \text{H}_2$ gaseous mixture. Micro-scratches were performed using a MST-CSEMX[®] system equipped with optical microscope, acoustic emission sensor and friction force sensor. Critical loads were measured to determine adhesion of TiN on substrate. Influence of various parameters such as scratching speed, loading rate, film thickness on critical loads were studied. Rockwell indentations, S.E.M. observations, electron probe microanalyses and chemical attack of the films were produced to identify and interpret damage mechanisms. Adhesion models previously propounded in literature were discussed and work of adhesion was evaluated.

MICROMECHANICAL ANALYSIS OF FAILURE MECHANISMS OF BN-LAYERS

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Layers consisting of the cubic phase of boron nitride (c-BN) exhibit out-standing mechanical properties for application as protective coatings. c-BN layers are usually prepared by the deposition of particles with comparatively high energies. Initially, a thin layer of the hexagonal phase of BN (h-BN) forms. At a certain h-BN thickness, the nucleation of c-BN starts owing to large compressive stresses in the layer. During further deposition, a c-BN layer with columnar grain structure grows. One problem in the manufacturing of c-BN coatings is the spontaneous spallation of thicker layers due to compressive stresses of more than 5 GPa.

The present theoretical study analyses the development of stress concentrations and possible failure mechanisms of the substrate/h-BN/c-BN layer system. Several mechanisms are investigated which can cause the formation of microcracks as well as the propagation of cracks resulting in layer delamination: (i) tensile stress concentrations due to the roughness of the h-BN/c-BN interface, (ii) the effect of h-BN inclusions in c-BN on the stress state, (iii) spatial stress variations due to competing growth of non-aligned adjacent grains, and (iv) a shear crack instability by microcrack interactions owing to the porosity in the layer. The complex stress fields connected with the above mechanisms have been calculated by means of the finite element method (FEM), taking into account the large elastic anisotropy of h-BN. The FEM calculations predict that the layer inhomogeneities listed above can cause large local tensile stresses of the order of 1 GPa. The stress concentrations are discussed in dependence on relevant geometric parameters as the h-BN and c-BN layer thicknesses, the interface roughness and the misorientation of adjacent grains. The proposed failure mechanisms are related to recent experimental observations of spontaneous c-BN layer spallation.

Mechanical Properties of Films, Coatings and Interfacial Materials
Poster Presentation

MECHANICAL PROPERTIES OF GRADED CU/W JOINTS

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Mechanical properties of Cu/W joints are investigated through a combination of numerical simulations and experiments. Residual stress distributions are manipulated by the strategic placement of interlayers, as shown by finite element modeling (FEM). Guided by the FEM studies, a wide variety of graded joint architectures have been produced by powder metallurgy methods. The fracture and deformation of these joints is examined and assessed with respect to the predicted residual stress distribution and fracture criteria for the interlayer materials.

CORRELATION BETWEEN REAL HARDNESS AND MICROSTRUCTURE AT THE SURFACE OF NITROGEN IMPLANTED STEELS

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In this paper, an hardness investigation associated to a microstructural characterization performed on Austenitic Stainless steel (ASS) and Ferritic Steel (FS) surface nitrided by ion implantation is reported. Implantation with N⁺ ions is aimed to improve wear resistance and fatigue behavior of a low carbon austenitic stainless steels and ferritic steels.

The surface of the austenitic X2CrNiMo17-13-2 and ferritic X1CrNiMoNb28-4-2 steel was nitrided by nitrogen implantation at doses of 0.3 - 10 10¹⁷ N⁺cm⁻², with an energy of 60 or 100 keV. The beam current range is from 1.5 μA cm⁻² to 3.5 μA cm⁻². The surface microstructure and hardness depend on the nitriding conditions and the bulk material. The experimental conditions determine the nature of nitride phases as well as the nitrogen redistribution below the surface [1]. This work aims to examine the effects of ion energy and current density during nitrogen implantation of steel.

X Ray Diffraction (XRD), Conversion Electron Mössbauer Spectroscopy (CEMS) were used to investigate the nitride phases resulting from nitrogen implantation. The implanted area is too thin to be directly measured from Vickers microhardness indentation. To determine the real hardness of the implanted zone (≤ 0.2μm) we examine our results by using the models of Jönsson and Hogmark [2] and improved by Iost and Bigot [3].

The Vickers microhardness, the microstructure and the nitrogen distribution at the surface of austenitic and ferritic steels are compared and discussed. Evolution of the phase formation is associated with chromium and nitrogen migration and segregation during the nitriding process. The formation of Cr₂N and of phases such as α'-martensite, γ_R-N enriched austenite, Fe₄N γ'-like structure, h.c.p. ε is detected depending on steel grade and nitriding conditions. The hardness of the implanted zone is higher than the hardness of the bulk material and is explained by the microstructure and the stresses due to the superficial treatment.

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**Cracking behaviour of magnetron sputtered chromium coatings
on a ductile steel : an experimental study.**

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Magnetron sputtered coatings are expected to improve the mechanical resistance to wear, corrosion or temperature of metallic parts in a lot of engineering applications. This resistance is known to depend on the main deposition parameters such as the substrate bias voltage, the target current, pressure and temperature. As-deposited coatings are frequently subjected to strong compressive residual stresses, which may reach -2GPa in Mo coatings and -4GPa in W coatings^{1,2}. These residual stresses in sputtered coatings can be explained by an "atomic peening"^{1,3} mechanism associated with the deposition of high energy atoms. This study aims at understanding the influence of such residual stresses on the cracking behaviour of magnetron sputtered chromium coatings deposited on a 35NCDV12 steel substrate.

Two 2µm thin coatings, resulting from different deposition conditions, have been investigated. The first one (coating A) was subjected to -5Volt substrate bias voltage, whereas the second one (coating B) suffered a -125Volt voltage. The residual stresses were evaluated according to Stoney's formula and the assumptions of plane stresses and elastic substrate, for a thickness ratio of 10^{-2} : these compressive stresses were estimated to -2.1GPa in coating B and to almost zero in coating A.

The mechanical behaviour of these coatings has been studied experimentally both at the global and local levels. Macroscopic tensile tests using strain gauges and an acoustic emission device were performed whereas scanning electron microscopy was used for crack observation as well as for local strain measurements. The latter was obtained by use of a microextensometer technique^{4,5} based on image analysis correlating high resolution images (4000x4000 pixels) of fiducial grids of 5µm path in the reference and deformed states.

In both coatings, mode I cracks are perpendicular to the tensile axis and do not reach the substrate. Coating A exhibit a classical⁶ brittle fracture high density (45 cracks/mm) crack pattern with cracks developed through the whole width of the sample. On the contrary, cracks are shorter in coating B; they do not cross the sample width and their density is lower (14 cracks/mm). A number of 1µm large microcavities are also present : they seem to play an important role in the crack formation and propagation.

A more detailed investigation has been performed on the coating B by comparing strain gauge measurements and local strain analysis : a representative surface element of about 500µm² has been determined and the local strain field at the coating surface has been examined at this scale. Some areas where no cracks are present have been shown to suffer measurable residual strains.

The significant differences in the cracking behaviour between both coatings must be correlated to the fact that, due to the strong compressive residual stresses in coating B, cracks appear after the substrate has entered the plastic regime, whereas it is still elastic in coating A. Further investigations are in progress in order to check the assumption of crack propagation in coating B developing by a percolation mechanism between the microcavities. More generally, we aim at optimizing the cracking behaviour of the coatings by mastering the influence of deposition parameters on the resulting residual stresses.

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FRACTURE AND RELAXATION OF THE INTERNAL STRESS OF A COATING SUBMITTED TO TENSION DURING THE DEFORMATION OF IT'S SUBSTRATE

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The evolution of the internal stress of a coating on a substrate system was investigated. The investigation was based on the results obtained when submitted the system to progressive pulling in a scanning electron microscope. These experiments allowed two sorts of analysis:

- first, the observation of the cracking properties. Indeed, when increasing the strain of the sample, the distance among two cracks of the coating tends to decrease to a constant value (crack saturation concept).

- Second, the determination of the internal stress evolution. At several level of strain, the sample was unloaded and submitted to x-ray diffraction analysis for in plane internal stress determination in the coating.

The main results of these analysis show for our investigated systems no debonding; but a particular evolution of the in plane internal stresses. Indeed, the coating shows a constant decreasing of the longitudinal component of the internal stress until almost a null value, while the transversal component remains at a constant value. These results are discussed with respect to intrinsic parameters of the system.

Mechanical Properties of Films, Coatings and Interfacial Materials
Oral Presentation

**PREPARATION AND PROPERTIES ENHANCEMENT OF SILICON CARBONITRIDE
FILMS USING REACTIVE MAGNETRON SPUTTERING**

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Silicon carbonitride ($\text{Si}(\text{C},\text{N})$) films were synthesized on Si (100) and metal substrates by reactive d.c. magnetron sputtering with Ar as the sputtering gas and N_2 as the reactive gas. The composition and properties of the films were studied with respect to the influences of the bias voltage applied to substrates, the deposition temperature, and the gas flow ratio of N_2 to Ar (or FN_2/FAr). The $\text{Si}(\text{C},\text{N})$ mechanical properties, hardness, tribological, and fracture, were observed to be highly depended on the processing conditions such as substrate temperature, the arrival ratios of ion to deposition atom, J_i/J_a , the negative bias voltage and FN_2/FAr . Under optimum conditions amorphous coatings with high wear resistance and harnesses as high as 40 GPa were prepared. The role of synthesis parameters on the structure, compositions, and mechanical properties will be discussed in detail.

INTERFACE FRACTURE TOUGHNESS MEASUREMENT FOR BIOACTIVE COATINGS OF BONE IMPLANTS

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Bioactive coatings for bone implants, especially those based on hydroxyapatite, are well known for many years and are more and more accepted in the medical community. However, delamination of these coatings has often been of major concern. A new electrochemical procedure for deposition of thin coatings is aimed to overcome these problems by providing a graded transition between titanium oxide and calcium phosphate.

Damage of the coating through delamination is always preceded by crack propagation parallel to the surface. This can be described by a fracture mechanical analysis using the energy release rate G for delamination. According to the fracture criterion, G has to be compared with the critical energy release rate G_c . Damage is avoided by keeping G below G_c . Therefore information on G_c has to be gathered.

The 4-point bending test after Charalambides has been modified such that G_c of the delamination crack of thin, brittle layers can be measured. The G required for crack propagation is obtained by attaching a stiffening layer to the coating. The experiment is analyzed by the finite element method (FEM). In order to measure G_c , one needs the critical load and the crack length. The latter is derived by FEM from the measured crack opening displacement.

Characterization of the bioactive coatings were carried out by means of Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). Fracture surfaces were examined by SEM and energy dispersive X-ray analysis (EDAX).

**ANALYSIS OF RESIDUAL STRESSES IN DIAMOND THIN FILMS DEPOSITED ON
SILICON BY HFCVD PROCESS**

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Diamond thin films were deposited on Si substrates by hot filament chemical vapor deposition(HFCVD) process at 1000jÉ for 10hours with various ratios of methane/hydrogen in precursor gases under total pressure of 40torr.

The deposited diamond thin films were annealed at 1150jÉ for 1 - 30min. in order to investigate the change of microstructure and residual stress during annealing treatment.

The residual stresses were measured by Raman spectroscopy, XRD and laser scanning curvature measurement methods. In XRD measurement, the residual stresses include errors due to the formation of texture in diamond thin films. In laser scanning curvature measurement method, the residual stresses include errors originated from the plastic deformation of Si substrate during the deposition at 1000jÉ. The Raman spectroscopy method was found to be the most reliable method for the characterization of residual stress in diamond thin film.

The microstructural factors of diamond thin films, i.e. grain size, surface morphology and non-diamond carbon content, were sensitively dependent on both the ratios of methane/hydrogen in precursor gases during deposition and the annealing treatment condition. The residual stresses were measured ranged -680 - -320MPa in as-deposited diamond thin films and were changed into ranged -1473 - -271MPa after annealing treatment.

The residual stress in diamond thin film was divided into two components of thermal stress and intrinsic stress. A new model was proposed to estimate the intrinsic stress based on the assumption that the non-diamond carbon is located at grain boundaries in diamond thin film and the residual stress is generated by grain boundary relaxation mechanism.

THE SPALLING MECHANISM OF THERMAL BARRIER COATING DURING OXIDATION AT 1100iÆ

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The lifetime of thermal barrier coatings (TBCs) depends largely on its thermal oxidation and thermal shock resistance, these properties are related to the preparing process of the coatings. At the present, the practical TBCs usually possess double-layer construction (bonding layer MCrAlY + ceramic layer Y-PSZ), the bonding and ceramic layers are prepared by plasma spray and /or EB-PVD processes. In this paper, the thermal oxidation resistance of a TBCs whose bonding layer NiCrAlY was prepared by vacuum arc deposition and the ceramic layer (8 wt.% Y₂O₃+92 wt.% ZrO₂) by EB-PVD without the supplement of O₂ during the deposition, was evaluated by a static oxidation test at 1100 iæ. The research emphasis are placed on the effect of different pre-treating methods including the untreated, sand-blasting and shot peening of the bonding layer surface on the adhesion of the ceramic layer and the failure mechanism due to the spallation of the ceramic layer . In addition, the phase transformation ion of the ceramic layer and the oxidation dynamics of the TBCs during the oxidation at 1100iæwere also investigated.

It has been found that adhesion of the ceramic layer of TBCs specimens decreased in the sequence of the untreated, shot peened and sand-blasting specimens. XRD results confirmed that the ceramic layer of the TBCs consisted of t(-ZrO₂ and c-zrO₂ two phases, and after the oxidation at 1100iæfor 100 hours, the phase structions of the ceramic layer did not change obviously. On all of specimens undergone thermal oxidation for 25, 50, 75 and 100 hours, no m-ZrO₂ phase was detected. SEM observation showed that the failure interface of TBCs on three kinds of the specimen represented quite different morphological characteristics which coincided with the fractures of spalled ceramic chips. SEM observation and XRD analysis also indicated that on the untreated specimen, the scale of the bonding layer surface consisted mainly of dense rich-(Al₂O₃ and spalled less; But on the sand-blasting specimen, the scale of the bonding layer surface was rich-Cr₂O₃ (H) and almost all of scale had spalled ; On the shot peened specimen , the situation was in the middle. EDS compositions of the failure interface and the backside of spalled ceramic chips suggested that in the order of the untreated, shot peened and sand-blasting specimens, Al and O contents on the failure interfaces increased gradually while Cr, Ni and Zr contents decreased gradually, but there were a large difference in these contents between the untreated and shot peened or sand-blasting specimens while such a difference between shot peened and sand-blasting specimens was very small. We concluded that in our experimental conditions, the spallation of the ceramic layer of TBCs during thermal oxidation at 1100iæ was caused by the oxidation of bonding layer NiCrAlY, the latter was related to the degradation of the protecting function of the scale on the bonding layer surface, which depended on the chemical composition and stress states of the bonding layer surface controlled by different pre-treating methods.

Keywords: thermal barrier coatings, spalling mechanism, pre-treatment

Poster

The preparation of thin films of yttrium aluminium garnet by solution routes

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The production of yttrium aluminium garnet (YAG) at low temperatures has previously been reported to require the gelling of sols derived from alkoxide precursors. In the present work it has been shown that segregation-free oxides in the alumina - yttria system may be obtained by the spray pyrolysis of aqueous solutions of nitrates, leading to the production of YAG at temperatures as low as 900 C. The crystallization product of the amorphous as-sprayed film is a hexagonal derivative of $YAlO_3$ that subsequently transforms to garnet. The flexibility of the solution route allows the possibility of producing coatings with gradients in composition across the thickness. The microstructure and indentation response of these films on alumina substrates will be described.

STRAIN EFFECT ON THE HETEROEPITAXY OF DIAMONDS GROWN ON SI

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Heteroepitaxial growth of diamond films is one of the critical issues in their potential applications to electronic devices. In this study, we present the strain effect on the heteroepitaxy of diamonds grown on silicon substrate in early stage. Diamonds were deposited on Si (111) at 970 °C by bell-jar type microwave plasma-enhanced chemical vapor deposition (MW-PECVD), using 0.4% methane in a balance of hydrogen. The substrates were ultrasonically treated in diamond suspension before diamond deposition. The microstructure and the strain of diamonds were characterized by using synchrotron x-ray scattering at beamline 5C2 at Pohang Light Source (PLS) in Korea. We found that diamonds were grown epitaxially on the silicon substrate with the epitaxial relationship of diamond(111)//Si(111) and diamond(11-1)// Si(11-1). Remarkably, the diamond(111) planes, in early growth stage, were very well aligned on the Si surface with the mosaic distribution of 0.03° full-width at half-maximum (FWHM), demonstrating a very high quality of diamond heteroepitaxy. We first report the strain of the diamond single crystal islands grown on Si in early stage as + 0.15 % in the out-of-plane direction. The strain was greatly relieved as the islands were so much grown that they began to come into contact one another. Further growth induced the gradual deformation of the islands, as demonstrated by the gradual tilting of the diamond(111) planes away from the silicon surface, resulting in deteriorating the epitaxial quality of diamond film. We suggest that this feature may be due to the internal stress accumulated on the diamond-silicon interface.

Measurement of interface stress and interface deformation in nanocrystalline Pd-H

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Due to the large interface to volume ratio, interfacial properties have important consequences for the overall macroscopic properties of nanocrystalline microstructures. In particular, interface stress, that is the derivative of the interfacial free energy with respect to strain tangential to the interface plane, results in stress in the bulk that influences the chemical equilibrium and phase stability [1]. Interfaces introduce additional degrees of freedom for deformation that have measurable consequences for the overall deformation of the microstructure. An example is 'stretch' [2], that is a deformation of the interface along the normal, modeling a change in the normal component of the mean interatomic distance. We present a combined dilatometry and diffraction study of hydrogen uptake in nanocrystalline Pd, that leads to measurement of the variation of interfacial stress and stretch with hydrogen concentration [3]. We suggest that interface stress not only changes the concentration in the bulk at equilibrium with the solute at a given chemical potential, but also the terminal solubilities and the critical temperature in the bulk alloy phase diagram.

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STUDIES OF STRENGTHENING MECHANISMS IN GOLD THIN FILMS ON SUBSTRATES

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The high strengths of gold thin films on silicon substrates have been studied to determine the contribution of thickness, passivation, and plastically-induced strain gradients on these properties. Wafer curvature/thermal cycling measurements were used to study the strengths of gold films ranging in thickness from 0.1 to 2.5 micrometers. We found that the room temperature stresses are related to the inverse of film thickness when the films are thicker than about 1 micrometer. Thinner films have stresses much lower than this relationship would predict, possibly due to diffusional relaxation effects near the free surface. The stresses were measured in subsequent thermal cycles after the films had been passivated. We found that the stresses are only minimally affected for films thicker than 1 micrometer, but thinner films were able to sustain much higher stresses, especially at elevated temperatures. This strengthening effect of the passivation is consistent with the shutting down of diffusion at the film's free surface.

In order to avoid strain gradients due to surface relaxation and passivation, our search for plastically-induced strain gradients focused on bare films thicker than 1 micrometer. Several x-ray diffraction techniques were used to investigate the elastic strains in these films at room temperature. A plane spacing vs. $\sin^2(\Psi)$ technique was used to find the average strain of these films. The results are consistent with the wafer curvature measurements. We also measured plane spacings as a function of penetration depth to construct depth-dependent plane spacing vs. $\sin^2(\Psi)$ plots. These data show that the stress-free lattice parameter varies with depth in these films but the residual elastic strain is independent of depth in the film. Finally, a novel technique for sample rotation was used to measure the plane spacings for a fixed set of grains as a function of penetration depth. Again, the stress-free lattice parameter is found to vary with depth, but no detectable gradient was observed. These results show that the high strengths of unpassivated gold films relative to the strength of bulk gold cannot be rationalized on the basis of strain gradients through the film thickness.

ON THE RESIDUAL STRESS AND MECHANICAL PROPERTIES OF THERMAL SPRAYED COATINGS: PROCESSING INFLUENCE AND ENGINEERING IMPLICATIONS

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Thermal spraying is a highly dynamic process resulting from rapid heating /accelerating of powder particles in a flame, followed by impact and rapid solidification of the droplets (splats). A splat resulting from the flattening of an individual droplet is the basic building block ("unit cell") of the thermal sprayed microstructure. Phase, microstructure and residual stress of the splats (intrinsic) and the integration of the splats (extrinsic) are affected by processing condition. The properties of a thermal sprayed deposit are directly related to this complex microstructure and stress state.

Residual stress is an important factor in thermally sprayed deposits which affects both processing and performance. Residual stresses in sprayed deposits results from large temperature excursions during processing and can be divided into two main components: quenching stress (associated with phase changes) and thermal mismatch stress (associated with expansion differences between substrate and deposit). In the high velocity deposition processes, an additional attribute is the peening effects on stress due to high velocity impact. In this study, the residual stresses in thermal spray deposits have been examined at the splat level, as an average in the deposit and through thickness gradients using curvature, x-ray diffraction and neutron diffraction methods. The results are assimilated based on processing influences and temperature excursions.

This presentation will also address other attributes of the sprayed coatings such as Young's modulus, hardness and anisotropy in fracture toughness of the coatings and the implications of these parameters on both processing and performance such as tribological response will be addressed.

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**EFFECTS OF NH₃ COLD PLASMA TREATMENTS ON THE ADHESION IN
ORGANIC COATING/ALUMINIUM SYSTEM**

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The adhesion of organic coatings on cold rolled aluminium alloy, treated using radio frequency plasma, have been tested via Electrochemical Impedance Spectroscopy. 8011 aluminium alloy have been used as a substrate, two types of solvent based organic coatings (i.e. epoxyphenolic and epoxypolyester) and one type of waterborne epoxyphenolic coating have been deposited on the aluminium. The plasma treatments were carried out in a parallel plate reactor using NH₃ as a gas feed and treatment time (i.e. 3-30-300 s). Results shown that plasma treatment produces strongly modification on metallic surface enhancing or decreasing adhesion at coating/substrate interface. We have shown that there is no correlation between contact angle and adhesion even if the liquid coating is used, then the practice to use water contact angle to forecast adhesion strength have no scientific support. Further cold plasma could be an effective clean technology to modify adhesion in painted aluminium systems and more data must be collected to find optimum experimental conditions. On the other hand less effective coatings can exhibit high performance if the adhesion at metal/coating interface is enhanced using an appropriate surface treatment, this is due to the role played by the solvent(s) and additives in the surface/ coating interactions.

DEPOSITION AND TEXTURE EVOLUTION OF IRON-NICKEL ALLOY THIN FILMS

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Fe-Ni alloy materials have been receiving great interests due to their useful thermomechanical and magnetic properties applicable to various electro-magnetic parts. It is well known that Fe-Ni alloys reveal much different physico-chemical properties as a function of composition. For example, Fe-36.5%Ni, which is referred to Invar, shows peculiar thermal anomalies, whereas other Fe-Ni alloys have prominent magnetic properties with increasing Ni content. Many attempts have been made to elucidate the properties related to the texture development of bulk Fe-Ni alloys. However, in spite of the increasing demand for thin films of Fe-Ni alloys in various industrial fields, the studies on Fe-Ni thin films are relatively limited. In the present work, we have successfully grown Fe-Ni alloy thin films on low carbon steel substrates employing sputtering method with a Fe-36.5%Ni target. Then the film properties were characterized focusing on the evolution of the textures in the films. The textures in the deposited films exhibited a dramatic change depending on the deposition conditions. A model explaining the texture evolution will be proposed and discussed in terms of the conventional models. Also we found that the Fe-Ni alloy deposition induced a significant decrease of thermal expansion in the film specimens, and confirmed that the adhesion between the film and the substrate was fairly good by a thermal cyclic fatigue test. The results will be discussed for the aspect of practical applications.

THERMO-MECHANICAL BEHAVIOR OF A Y3Al5O12 BOUNDARY

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The behavior of Y3Al5O12 (YAG) has recently been of interest in composite materials for high temperature applications because of its high strength, high corrosive resistance and low creep rate. Of particular interest is the structural behavior of YAG at elevated temperatures. The thermo-mechanical behavior of YAG is examined by conducting thermal grooving experiments on grain boundaries in addition to elevated temperature boundary sliding experiments. Model grain boundaries were synthesized by diffusion bonding two YAG single crystals to form an extremely flat S3 symmetric tilt boundary. Compression tests in air up to 1750°C resulted in boundary sliding. Grain boundary thermal grooving was measured with AFM to assess the role of diffusion in grain boundary sliding. SEM and TEM were used extensively to characterize the boundary and study the role of dislocations in the sliding process.

**DEVELOPMENT OF A FUNCTIONALLY GRADED CHROMIUM NITRIDE
COATING DEPOSITED BY CATHODIC ARC EVAPORATION FOR WEAR
RESISTANT APPLICATIONS**

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Surface modification of dies for forming operations can dramatically improve their lifetimes. However, it is clear that coatings developed for one type of operation are not transferable to another operation. For example, a coating developed for cutting operations may not necessarily be used successfully for forming operations. Chromium nitride has been researched for its good wear resistant properties, especially on non-ferrous materials and soft substrates, where more brittle titanium-based coatings fail rapidly.

It is noted that the highest stress concentration of a loaded coating-substrate system lies at the interface. Therefore, the coating deposited must have high interfacial adhesion to resist stress induced delamination. Furthermore, since a compressive coating stress will cause a tensile stress in the substrate, or vice versa, coating stress must be minimized to prevent the stress concentrations in the coating / substrate from degrading film performance. It is also known that increasing the film hardness can decrease adhesive wear by minimizing local plastic deformation due to high contact pressure between sliding surfaces.

The author's research done to date on Cr-N has yielded a fundamental understanding of the parameter-property relationships and interdependencies. However, it was found that hardness and adhesion exhibit a negative correlation. This presents the problem in that both properties must be present for the film to perform well as a wear resistant coating.

To overcome these experimental results, a compositionally graded architecture for the chromium nitride coating system is hypothesized. Several methods are discussed including the usage of varying exponents in a power law function, fundamental observations and high voltage pulsed bias (Hyper-Ion™, ISM Technologies, Inc.) deposition. Microhardness, residual stress, critical load, wear resistance and microstructures have been characterized and are reported. Drilling tests done on Cr-N coated drill bits has also been performed and the results are discussed.

ANALYSIS OF BONDING INTERFACE STRENGTH OF STRUCTURES REALIZED BY WAFER BONDING

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Silicon wafer bonding is increasingly used in the fields of microelectronics [1], in particular for achieving materials such as SOI structures. Meanwhile, any applications of these bonded structures remain strongly dependent of the chemical nature of the surfaces, of their hydrophilicity, of their microroughness and also of their particle contamination.

Previous to the bonding, the surface hydrophilicity is characterized by contact angle measurement, the surface microroughness by Atomic Force Microscopy (AFM) and the particle contamination by light pattern defect density determination thanks to a surfscan from Tencor. Then, bonding is qualitatively evaluated by several methods including IR transmission and acoustic microscopy.

In order to quantify the bonding strength, the usual technique is the crack opening method [2] where a blade is introduced at the bonding interface. The length of unbonded area is connected to the bonding energy. This method allows us to correlate the bonding energy to the thermal treatment used to strengthen the bonding according to the cleaning process (figure1). In the same way, bonding energy can be checked versus microroughness surface [3].

But, in order to better understand the wafer bonding mechanism and to characterize the reliability of the structures, bending and pulling tests are performed. Special grips and specific samples are used. These conditions allow us to test the bonded structures under various loading conditions.

The experimental results, in term of stresses and rupture energies, are discussed with respect to the microstructural observations of the cracked and debonded surfaces.

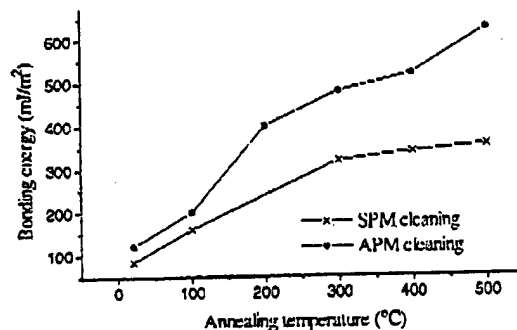


Figure 1 : Cleaning effect on bonding energy after a 30 mn annealing using the crack opening method

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**DEFORMATION BEHAVIOR OF A GRADED NICKEL- Al_2O_3 COMPOSITE MEASURED
WITH PHASE SHIFTED MOIRÉ INTERFEROMETRY**

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Phase shifted moiré interferometry (PSMI) was used to measure the deformation of a discretely layered functionally graded material (FGM) loaded in compression. A wedge loaded notch geometry was also tested. Finite element analysis (FEA) predicted thermal residual stresses and subsequent mechanical loading displacements. A ceramic-metal-ceramic FGM with discrete layers was formed from elemental powders. Two 8mm thick 100% Al_2O_3 layers were joined with (9) 1mm thick interlayers. Layer composition transitioned from 80% Al_2O_3 to 100% Ni in the center layer. High residual tensile stresses near the edge of the pure Al_2O_3 /80% Al_2O_3 interface (as predicted by FEA) made fabrication and specimen sectioning difficult. PSMI was used to measure the layer displacement evolution for increasing compressive loads under the influence of an initial residual stress field. Displacement was also measured for an FGM with a wedge loaded notch machined parallel to the 20% Al_2O_3 /pure Ni interface. Experimental displacement fields are directly compared to the FEA.

**MECHANICAL PROPERTIES OF GALVANIZED COATINGS:
CHARACTERIZATION, MODELLING AND APPLICATION TO SCC BEHAVIOR**

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Zinc coatings are constituted by heterogeneous assembly of phases of which the mechanical properties greatly differ from each other. Thermal strains resulting from large differences between thermal expansion coefficients are partially relaxed by the formation of a crack network in the δ 1-phase. In order to model this phenomenon, hardness Hv, thermal expansion coefficient, and toughness K_{IC} are determined. The δ 1-phase which is the most brittle phase, is proved to constitute the most critical constituent in the coating. Taking into account the critical energy for crack propagation G_{IC}, the conditions for crack network formation and propagation have been modelled comparing the expansion of the plastic zone in the δ 1-phase, in the substrate and at the δ 1-substrate interface.

Hot dip galvanizing is a surface treatment consisting in a zinc alloyed coating, known to be very efficient for protecting the steel against corrosion. This study shows the susceptibility to the stress corrosion cracking (SCC) of a galvanized steel at free potential associated with a ductile to brittle fracture transition. Electrochemical considerations and micrographical studies allow to give to the coating an important role in the crack initiation and hydrogen embrittlement (HE) mechanisms under specific experimental conditions.

Considering the low-toughness of the δ 1-phase leading to cracks formation, under loading, mechanical damage may fastly occurs in the coating and zinc corrosion is related to the lower potential of the δ 1 phase. Thus a crack can start to localize at the crack tip the hydrogen effects such as the cation hydrolysis reaction. Since the crack reaches the steel, zinc corrosion speeds up (sacrificial anode) with hydrogen release through the higher zinc hydrogen overvoltage than the iron one. When the strain rate is slow, white dust (Zn(OH)₂) can cover cracks, isolating them partially to the bulk environment. Then both oxygen reduction on the specimen surface and hydrogen reduction at the crack tip, can simultaneously occur. Moreover hydrogen enters in steels and HE is observed.

Mechanical Properties of Films, Coatings and Interfacial Materials
Oral Presentation

**TWO-DIMENSIONAL FINITE ELEMENT ANALYSIS OF THE STRESSES, FRACTURE
AND DELAMINATION DURING THE INDENTATION OF HARD ELASTIC FILMS ON
ELASTIC-PLASTIC ALUMINUM SUBSTRATES**

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In this work, the wear behavior of hard films on soft substrates was studied based on the finite element analysis of the indentation with normal forces. As an attempt to simulate situations close to the ones seen in practice, defects were considered during the preparation of the mesh, both in the film and at the interface. Three steps were considered during the loading sequence applied in the models. Initially, the deposition (intrinsic) stresses were introduced and, later, a temperature drop was applied to incorporate the thermal residual (extrinsic) stresses. In the third step, an indentation with a rigid spherical indenter was applied. The influence of film thickness and elastic modulus was studied based on the radial and shear stresses that develop at the film surface and at the film/substrate interface. Results were also compared to ones obtained for the aluminum substrates and no film.

INDENTATION CHARACTERIZATION OF POLYMER COATINGS

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Sacrificial polymeric thin films are often used as protective overcoats. Adhesion of the polymer is governed by the yield strength as well as the modulus of the coating. We have investigated a nanoindentation and imaging technique to assess the yield strength and modulus as well as the adhesion of the system. The yield strength is determined via a plastic-zone model, which is applicable for materials which show an elasto-plastic response to indentation. The modulus employs an elasto-viscous analysis which utilizes the creep or relaxation response of the system. Both measurements and an annular plate adhesion model developed by Thouless and further investigated by Ritter et al. lead to quantifying the adhesion of the polymer to the substrate in terms of the strain energy release rate.

The thin film systems used are a 16 μm styrene-acrylate electropaint on aluminum and a 1.7 μm epoxy on aluminum. For the styrene-acrylate coating, the effect of processing parameters such as crosslink density as well as filler addition are discussed in terms of adhesion as well as yield strength and modulus. For the thinner epoxy coating the effect of a 100 nm interlayer on the adhesion of epoxy to aluminum is also discussed in terms of the adhesive strength. With the addition of the interlayer the epoxy could not be deadhered from the aluminum, i.e. no interfacial crack could be initiated. The contribution of the interlayer was evaluated using an adhesive strength model originally developed by Matthewson, which is also based on the yield strength of the coating. Instruments used in this study were a Nanoinstrument Nanoindenter II, a Perkin Elmer Dynamic Mechanical Analyzer and a Micromechanical Testing Instrument developed by IBM as well as a Digital Instruments Atomic Force Microscope.

ON THE MEASUREMENT OF RESIDUAL STRESS BY LOAD AND DEPTH SENSING INDENTATION

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The finite element method was used to determine whether load and depth sensing indentation with spherical indenters may be useful in the measurement of residual stresses in materials. The spherical indentation process for a wide range of elastic/ideal-plastic materials to which compressive and tensile biaxial stresses were applied was simulated using standard finite element techniques. The elastic moduli and yield stresses of the materials were varied systematically to model the behavior of a wide variety of metals and ceramics, with residual stress levels varied from zero up to the yield stress. All three indentation regimes - elastic, elastic-plastic, and plastic - were examined, with emphasis given to the elastic and the early part of the elastic-plastic regimes, where differences in the load-displacement characteristics caused by residual stress were found to have a particularly significant effect. Systematic examination of the relationships among residual stress, contact pressure, pile-up height, and elastic recovery revealed a simple, measurable indentation parameter which correlates well with the residual stress. Using this parameter, an experimental technique is proposed by which residual stresses can be estimated from measurements of the load at the elastic to elastic-plastic transition, the yield stress, and the elastic modulus of the material, all of which can be determined by load and depth sensing indentation methods. Based on a critical examination of the technique by finite element simulation, the technique appears promising. Additional simulations were performed to study the feasibility of using the method to determine residual stress in different types of materials. It was found that residual stress in elastic/plastic materials with elastic modulus to yield stress ratios ranging from 10 to 1000 can be measured using experimental observations made with spherical indenters with diameters varying from about 10 micrometers to about 5 mm, respectively.

Surface hardness modification produced by hydrogen charging

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Hydrogen embrittlement has been studied for a long time but the mechanism has not yet explained clearly. The difficulty lies in the fact that the cause of embrittlement can change from one alloy to another. In addition, a large part of the reaction take place at the surface, especially at the beginning of the process. Thus analysis of the surface should provide useful information about the role of hydrogen in the process.

Microhardness measurements were carried out on austenitic stainless steels (Cr-19 Mn-19 polycrystals and Cr-18 Ni-14 Mo-2 single crystals with or without nitrogen addition) with a view to account on surface modifications produced by hydrogen diffusion.

To begin with, a composite model was developped to characterize hardness of thin films. It is shown that the apparent increase in Vickers Hardness Number resulting from hydrogen charging is an artefact and corresponds in fact as an increase of residual compressive stress. The thickness of the modified surface layer was calculated by our model and verified by Scanning Electron Microscopy on fractured surfaces obtained by tensile test. The nitrogen addition seems to promote the hydrogen diffusion in a small extend.

Moreover, it can be shown that hydrogen embrittlement induced a lower fracture toughness of the specimen surface. The K_{IC} values obtained from the crack pattern induced by the Vickers indenter are as low as 1-2 MPa \sqrt{m} and depend on the applied load wich is consistant with compressive residual stresses.

Degassing of hydrogen charged specimens decreases the residual stresses and the Indentation Size Effect i.e. the variation of VHN with the applied load.

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MECHANICAL PROPERTIES AND RESIDUAL STRESSES IN AlN FILMS
PREPARED BY ION BEAM ASSISTED DEPOSITION

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Aluminum nitride (AlN) thin films were prepared on silicon single crystal substrates by ion-beam assisted deposition method, and the influence of the nitrogen ion beam energy on mechanical properties and residual stresses was studied by changing the nitrogen ion beam energy from 0.1 to 1.5 keV. Mechanical properties were examined by a nano-indentation method. Residual stresses were evaluated by film curvature measured by an optical cantilever system. The films show elastic behaviors during loading and unloading processes, but the residual depth after the unloading process increases with increasing the ion beam energy, resulting in decreasing in the returned energy ratio. All the films are found to be in compressive stress state and the values of the stress decrease with increasing the ion beam energy. Decreasing tendency is also observed in the relationship between the ion beam energy and film hardness. So as to study the effect of thermal treatment on relaxation of residual stresses, the films were annealed in nitrogen atmosphere at 500 °C, and it is found that the stress reduction rate, which is defined as the ratio of the residual stress after annealing to that before annealing, is about 0.8 and 0.2 for the films prepared with the ion beam energy of 0.2 keV and 1.5 keV, respectively. These results suggest that rearrangement of AlN occurs readily in the films prepared with high ion beam energy. It is proposed that the mechanical properties and residual stresses are closely related with each other and they can be controlled by the ion beam energy.